

MTL TR 88-41

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# STRUCTURES AND PROPERTIES OF MAGNESIUM BASE COMPOSITES

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MATERIALS PRODUCIBILITY BRANCH

December 1988

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 88-41	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle)  STRUCTURES AND PROPERTIES OF MAGNESIUM BASE COMPOSITES		5. TYPE OF REPORT & PERIOD COVERED
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s)  Ernest S. C. Chin		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 ATTN: SLCMT-MEM		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS  D/A Project 1L162105AH84
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE December 1988
		13. NUMBER OF PAGES 9
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report)  Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES Published in Magnesium Developments, 1988, p. 46-52. A selection of papers from a program presented at the World Materials Congress, McCormick Place, Chicago, Illinois, held September 26, 1988.		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Composites , Metal matrix, Magnesium	Boron carbide , Silicon carbide, Alumina ,	Graphite , Fibers, Particulates . (Grs) ←
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  (SEE REVERSE SIDE)		

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## ABSTRACT

Chronic needs for higher performance in present and future military and commercial systems mandate improvements in material properties. High stiffness, high strength, and low density are among the material properties necessary to achieve future system performance goals. Such requirements can no longer be met using conventional metal alloys. Magnesium matrix composites are among the candidates to fulfill the aforementioned requirements. Ceramic fiber, particulate, and whisker reinforced magnesium composites have demonstrated significant improvements in specific stiffness and specific strength over the monolithic matrix alloys. Magnesium matrix composites can also compete with high strength aluminum alloys and other metal matrix composites for high performance and weight critical applications. However, magnesium composites are still relatively expensive and are still in the developmental stage. Six magnesium composites were studied: cast 55v/o continuous  $Al_2O_3$  fiber/ZE41A Mg; cast 40v/o continuous graphite fiber/ZE41A Mg; cast 40v/o continuous graphite fiber/AZ91C Mg; PM extruded 20v/o  $B_4C$  particulate/AZ61A Mg; PM extruded 20v/o SiC particulate/ZK60A Mg; and PM extruded 20v/o  $\alpha$ -SiC whisker/ZK60A Mg, cast 55v/o continuous  $Al_2O_3$  fiber/ZE41A Mg. Both tensile and fracture toughness results on each of the composite systems will be presented. The nature of the reinforcement-matrix interfacial bond was revealed through detailed transmission electron microscopy. The effects of the interface as related to the tensile and toughness properties will be discussed.

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## STRUCTURES AND PROPERTIES OF MAGNESIUM BASE COMPOSITES

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### INTRODUCTION

Magnesium is recognized as an engineering alloy for a wide range of weight sensitive applications. Higher strength and better corrosion resistance magnesium alloys are constantly being developed. The incorporation of hard phases as reinforcements to a magnesium matrix can result in enhanced specific strength and specific modulus as compared to the monolithic materials. Consequently, magnesium composites can compete with other monolithic engineering alloys such as aluminum and steel in weight critical applications.

Metal matrix composites have been the subject of intense research and development within the past ten years. However, to a relatively conservative engineering community, any novel material must first prove itself to be reliable, reproducible, and economical before being accepted. This requires the availability of a comprehensive material property data base compiled through detailed characterization of state-of-the-art materials.

Understanding the interdependent factors that correlate mechanical properties with microstructures and processing is one of the keys to material optimization. This is especially true for metal matrix composites. Each component in the composite microstructure plays significant role in its performance. The effects of reinforcement distribution, size, and interfacial reaction zone on composite strength and toughness are some of the many issues being investigated.

There are currently two types of magnesium composites: continuous fiber reinforced and particulate/whisker reinforced magnesium. In continuous fiber reinforced composites, the matrix serves as a binder for the load bearing filaments. Properties of continuous fiber reinforced composites rely on the filament properties and the capability of the fiber/matrix interface to transfer load. Theoretical properties of continuous fiber reinforced composites can be calculated by the rule-of-mixtures[1]. In particulate and whisker reinforced composites, the primary strengthening mechanism is retardation of dislocation movements by the fine dispersion of reinforcement. Numerous theoretical models relating dispersion mean-free-path, interparticle spacing, and other factors with mechanical properties have been proposed[2].

Six composites were studied for this paper:

1. 55 volume percent(v/o) unidirectional continuous alumina fiber/ ZE41A magnesium( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A);
2. 40 v/o unidirectional continuous P-55 graphite fiber/ZE41A magnesium (Gr/ZE41A);
3. 40 v/o unidirectional P-55 graphite fiber/AZ91C magnesium (Gr/AZ91C);
4. 20 v/o boron carbide particulate /AZ61A magnesium(B<sub>4</sub>C/AZ61A);
5. 20 v/o silicon carbide particulate /ZK60A magnesium(SiC/ZK60A);
6. 20 v/o silicon carbide whisker/ZK60A magnesium(SiC<sub>w</sub>/ZK60A).

The  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A was processed by method of molten liquid metal infiltration [3]. All the Gr/Mg composites in this study were consolidated by investment casting [4]. The graphite fibers were pretreated with an oxide coating [5] through oxidation of an organometallic solution on the filament surfaces[5] prior to casting to enhance wetting and interfacial bonding.

Particulate and whisker reinforced magnesium composites were processed through powder metallurgy(PM) techniques. The particulates or whiskers were blended with the matrix powder, canned, degassed, hot pressed, and extruded[6]. The blending of metal powders and reinforcements was a key process where segregations of the hard phases in the composite microstructure must be avoided. The B<sub>4</sub>C/ZK60A studied was a 3" diameter extrusion subjected to a 4:1 cross section reduction. Both the SiC/ZK60A and SiC<sub>w</sub>/ZK60A were 2" diameter 13:1 extrusions. Further heat treatments were performed to strengthen the matrix alloy. The SiC/ZK60A, and SiC<sub>w</sub>/ZK60A studied were in the T6, solution heat treated and artificially aged, condition. The B<sub>4</sub>C/AZ61 was studied in the as extruded (F) condition.

### MATERIAL CHARACTERIZATION TECHNIQUES

In this study, tensile properties, toughness behavior, fracture characteristics, and microstructures were determined for each individual composite. The relationship between microstructure and properties were explored through observations. Longitudinal and transverse tensile properties were determined with tapered "dog bone" specimens sectioned from 1/2" thick  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A, 1/4" thick

Gr/ZE41A, and 1/4" thick Gr/AZ91C plates. Round button head and threaded specimens were tested in the radial and extrusion direction for B<sub>4</sub>C/AZ61A, SiC<sub>p</sub>/ZK60A, and SiC<sub>w</sub>/ZK60A. Charpy V-notch specimens were sectioned from 1/2" thick  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A, Gr/ZE41A, and Gr/AZ91C plates. Similarly, Charpy V-notch specimens were also sectioned from the 3" diameter B<sub>4</sub>C/ZK60A, 2" diameter SiC<sub>p</sub>/ZK60A, and 2" diameter SiC<sub>w</sub>/ZK60A extruded rods. Relative toughness of each composite was established through comparison of K<sub>IC</sub> values obtained from slow bend testing of the Charpy specimens according to ASTM E399 test method without precracking. Fracture morphologies of each of the composites were determined through detailed scanning electron microscope (SEM) observations. Microstructural and interfacial characterizations were performed with metallography and transmission electron microscope (TEM) techniques.

## RESULTS

### MICROSTRUCTURES

#### Reinforcement Size and Distribution

The  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A fibers are 20  $\mu$ m diameter polycrystalline filaments[7], whereas the graphite P-55's are 10  $\mu$ m diameter pitch base carbon fibers[8]. Metallography and SEM study of the polished microstructure showed relatively uniform fiber distribution in both the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A, Gr/AZ91C, and Gr/ZE41A composites (see Figure 1). Matrix rich zones separating laminate layers were observed in 1/4" thick plates and were prevalent in 1/2" Gr/Mg plates.

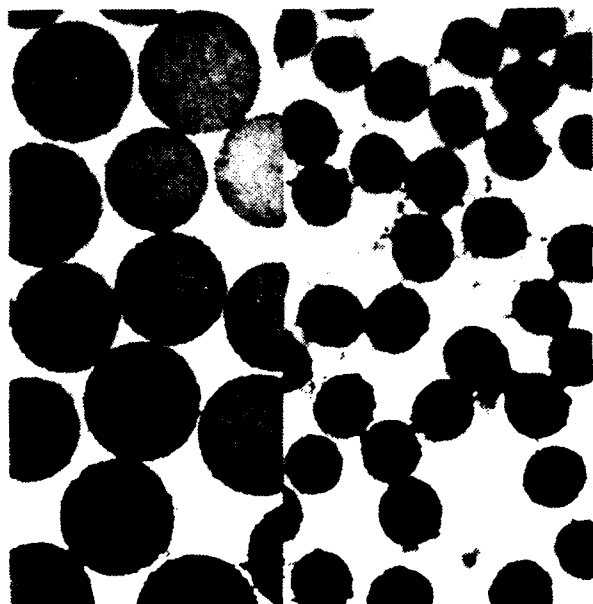


Figure 1. Optical Micrograph of a Typical Transverse Cross Section in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A (Left) and Gr/AZ91C (Right). (1000X)

The whisker and particulate reinforcements varied in size and geometry throughout each of the dispersion strengthened composites. The B<sub>4</sub>C particulates are polygonal in nature with a mean diameter of 10  $\mu$ m (see Figure 2). The SiC particulates were fine spherical powders less than 5  $\mu$ m in diameter (see Figure 3). The SiC whiskers were single crystals and had a mean diameter of 0.6  $\mu$ m and an aspect ratio of 10:1 (see Figure 4). All the whiskers were aligned in the extrusion direction. The B<sub>4</sub>C, SiC<sub>p</sub>, and SiC<sub>w</sub> reinforced Mg composites showed pockets of matrix rich zone in the transverse direction (see Figure 3). Larger unreinforced matrix rich zones were found in SiC<sub>p</sub>/ZK60A than in SiC<sub>w</sub>/ZK60A. Sporadic clusters of reinforcement were found in all of the composites.

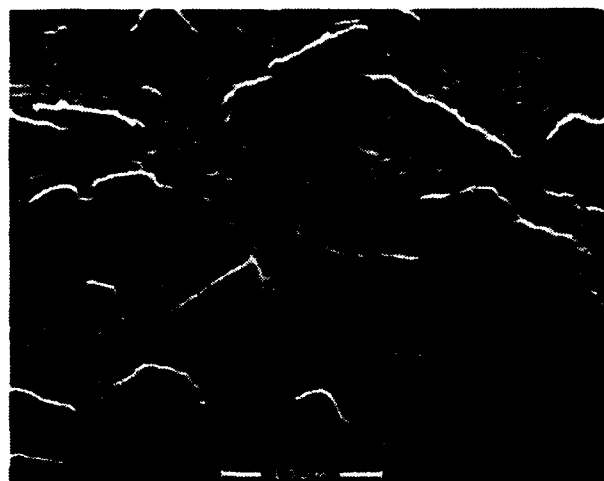


Figure 2. SEM Micrograph of a Typical Polished Transverse Cross Section in B<sub>4</sub>C/AZ61A.

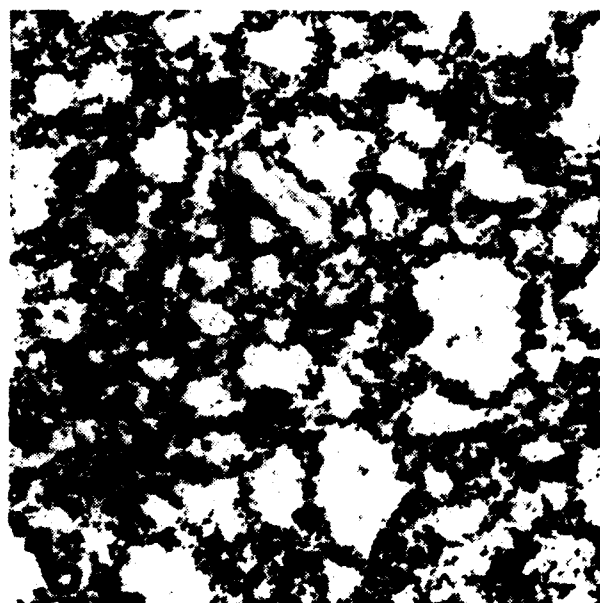


Figure 3. Optical Micrograph of a Typical Transverse Cross Section of SiC<sub>p</sub>/ZK60A. (1000X)

## Interface

Previous studies on  $\alpha\text{-Al}_2\text{O}_3/\text{ZE41A}$  identified a fine grained spinel ( $\text{MgAl}_2\text{O}_4$ ) phase separating the fiber surface and the magnesium matrix [9] (see Figure 5). In Gr/ZE41A composite, TEM had revealed large silicon and rare earth rich oxide particles at the interface (see Figure 6), whereas in Gr/AZ91C, a thin layer of amorphous silicon oxide and a layer of magnesium oxide defined the interfaces (see Figure 7). The silicon oxide layer remaining between the fiber surface and the reacted magnesium oxide layer was part of the original coating deposited by oxidation of an organometallic solution. The  $\text{B}_4\text{C}/\text{AZ61A}$  particulate/matrix interface showed a fine thin reaction film (see Figure 8). In both the  $\text{SiC}_p$  and  $\text{SiC}$  reinforced ZK60A, no observable interfacial reaction zone was detected (see figure 8 & 9).



Figure 4. Optical Micrograph of  $\text{SiC}_p/\text{ZK60A}$  Longitudinal Cross Section.(1000X)



Figure 5. TEM Micrograph of Transverse Cross Section of  $\alpha\text{-Al}_2\text{O}_3/\text{ZE41A}$  Interface.(38,000X)



Figure 6. TEM Micrograph of Transverse Cross Section of Gr/ZE41A Interface.(330,000X)



Figure 7. TEM Micrograph of Transverse Cross Section of Gr/AZ91C Interface.(100,000X)

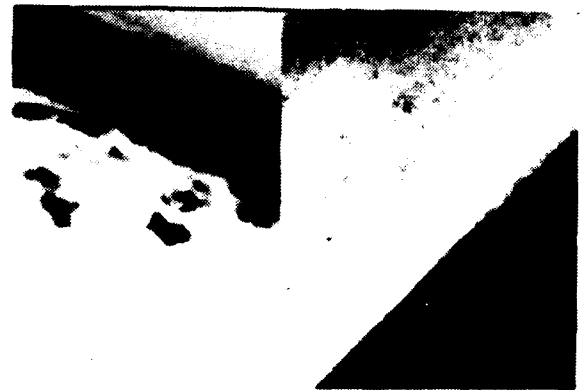


Figure 8. TEM Micrograph of Transverse Cross Section of  $\text{B}_4\text{C}/\text{AZ61A}$ (Left) and  $\text{SiC}_p/\text{ZK60A}$ (Right) Interface.(330,000X)



Figure 9. TEM Micrograph of Transverse Cross Section of SiC<sub>p</sub>/ZK60A Interface. (100,000X/Left, 330,000X/Right)

#### Matrix

Except for Gr/AZ91C where only dislocations were observed, substantial amount of matrix precipitation was found in all of the composites. The primary precipitation was large spherical MgZn and rod like MgZn' (see Figure 10). The MgZn precipitates were typically found at twin and grain boundaries whereas the MgZn' resided within each grain in a preferred orientation. The major difference between each composite were the size and distribution of these precipitates. The as-cast  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A had greater quantity and finer MgZn' in the matrix than the Gr/ZE41A. The B<sub>4</sub>C/AZ61A matrix was densely populated with matrix and grain boundary Mg-Al-Zn precipitates.



Figure 10. TEM Micrograph of MgZn' and MgZn Precipitation in SiC<sub>p</sub>/ZK60A. (59,000X)

#### MECHANICAL PROPERTIES

A summary of the tensile and toughness properties of each of the composites is found in Table I, II, and III. The tensile moduli of the continuously reinforced composites are superior to those of the dispersion reinforced composites. As expected in continuous reinforced composites, there is a significant drop in tensile and toughness properties from the longitudinal direction to the transverse direction. A marked decrease in properties from the 1/4" plate to the 1/2" plate in

the Gr/AZ91C is also noted. Although  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A has a higher density than Gr/AZ91C, it has better overall tensile and toughness properties in the longitudinal and transverse directions. In dispersion hardened composites, both SiCw/ZK60A and SiC<sub>p</sub>/ZK60A composites demonstrated better strength and modulus than the B<sub>4</sub>C/AZ61A. The highest strength of all the dispersion composites tested is SiC<sub>p</sub>/ZK60A in the longitudinal direction. The longitudinal tensile modulus in SiC<sub>p</sub>/ZK60A are identical, but both are higher than the B<sub>4</sub>C/AZ61A. In the transverse direction, better strength and modulus was attained with SiC<sub>p</sub>/ZK60A than with SiCw/ZK60A. The best toughness value attained with all the tested composites is demonstrated with B<sub>4</sub>C/AZ61A.

Table I. Longitudinal Tensile Properties

Material	0.2%Yield	UTS(ksi)	Modulus(Msi)
55v/o			
$\alpha$ -Al <sub>2</sub> O <sub>3</sub> /ZE41A	-	77.2	31.3
(1/2" Plates)		sd=2.5 (3%)	sd=0.3 (<1%)
40v/o			
Gr/AZ91C	-	85.0	26.7
(1/4" Plates)		sd=8.0 (9%)	sd=4.0 (15%)
(1/2" Plates)	-	62.0	17.8
40v/o			
Gr/ZE41A	-	40.5	29.6
(1/4" Plates)		sd=6.0 (15%)	sd=2.0 (7%)
20v/o			
B <sub>4</sub> C/AZ61A-F	38.5	47.9	8.5
(3" Dia.Extr.)	sd=0.7 (2%)	sd=1.2 (3%)	sd=0.4 (5%)
20v/o			
SiCp/ZK60A-T6	57.9	67.0	10.0
(2" Dia.Extr.)	sd=0.5 (<1%)	sd=0.9 (1%)	sd=0.5 (5%)
20v/o			
SiC <sub>p</sub> /ZK60A-T6	64.9	83.7	10.1
(2" Dia.Extr.)	sd=3.0 (5%)	sd=3.0 (4%)	sd=1.0 (10%)

\*sd=Standard Deviation

Table II. Transverse Tensile Properties

Material	0.2%Yield	UTS(ksi)	Modulus(Msi)
55v/o			
$\alpha$ -Al <sub>2</sub> O <sub>3</sub> /ZE41A	-	33.4	15.1
(1/2" Plate)		sd=0.3 (<1%)	sd=0.4 (3%)
40v/o			
Gr/AZ91C	-	6.5	4.1
(1/4" Plates)		sd=1.5 (23%)	sd=0.3 (7%)
(1/2" Plates)	-	1.1	3.5
40v/o			
Gr/ZE41A	-	2.7	3.6
(1/4" Plates)		sd=0.7 (26%)	sd=1.0 (28%)
20v/o			
SiC <sub>p</sub> /ZK60A	39.3	49.6	8.5
(2" Extr.)	sd=2.5 (5%)	sd=1.1 (3%)	sd=0.4 (5%)
20v/o			
SiC <sub>p</sub> /ZK60A	50.3	58.9	9.2
(2" Extr.)	sd=0.8 (2%)	sd=7.6 (13%)	sd=0.6 (6%)

\*sd=Standard Deviation



Table III. Fracture Toughness Properties

Material/Orientation	$K_{I0}$	
55v/o $\alpha$ -Al <sub>2</sub> O <sub>3</sub> /ZE41A		
L-T	13.4	sd=0.3(2%)
T-L	10.3	sd=1.6(16%)
40v/o Gr/AZ91C		
L-T	2.3	sd=0.1(6%)
T-L	0.08	
40v/o Gr/ZE41A		
L-T	1.21	sd=0.1(10%)
T-L	0.22	sd=0.05(23%)
20v/o B <sub>4</sub> C/AZ61A		
L-R	17.4	sd=0.6(3%)
20v/o SiC/ZK60A		
L-R	16.5	sd=0.5(4%)
R-L	12.1	sd=0.9(8%)
20v/o SiC/ZK60A		
L-R	14.7	sd=0.6(3%)
R-L	12.3	sd=0.5(4%)

\*sd= Standard Deviation

## FRACTOGRAPHY

Fracture morphologies from the tested tensile and toughness specimens were similar. The fracture morphology of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was typical of a well bonded fiber reinforced composite in the longitudinal direction (see Figure 11) and fiber splitting was apparent in the transverse direction. In the Gr/AZ91C, fracture occurred along the fiber surface and the silicon oxide coating interface (see Figure 12). Longitudinal fracture surfaces resembled those identified in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A. Fiber splitting was also characteristic of the transverse fracture morphology in Gr/AZ91C (see Figure 13). Rare earchth oxide and matrix debonding was the failure mechanism observed in Gr/ZE41A (see Figure 14). The longitudinal fracture surface was planar and characteristic of a brittle failure. In B<sub>4</sub>C/AZ61A, fracture typically initiated at a surface or internal flaw. Fracture initiation defects were often agglomerates of reinforcements or oxides. The general fracture morphology was composed of ductile fracture through the matrix connecting particles which failed in cleavage (see Figure 15). In SiC/ZK60A, micro-void coalescence and decohesion of particulates were the primary failure mechanisms (see Figure 16). A similar failure mode was observed in SiC/ZK60A with whisker pull-outs as an added fracture mechanism (see Figure 17).

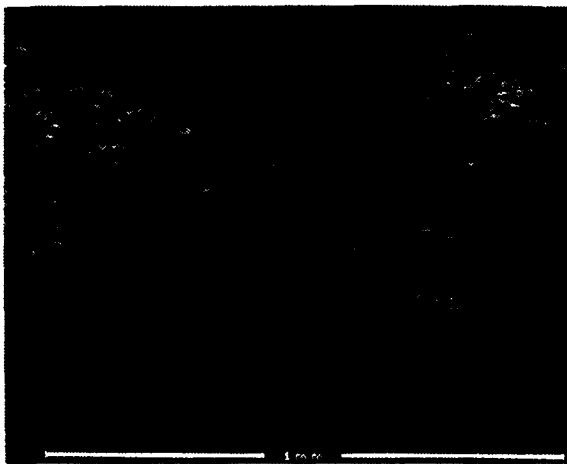
Figure 11. SEM Micrograph of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A Longitudinal Fracture Surface.

Figure 12. SEM Micrograph of Gr/AZ91C Longitudinal Fracture Surface.



Figure 13. SEM Micrograph of Gr/AZ91C Transverse Fracture Surface.

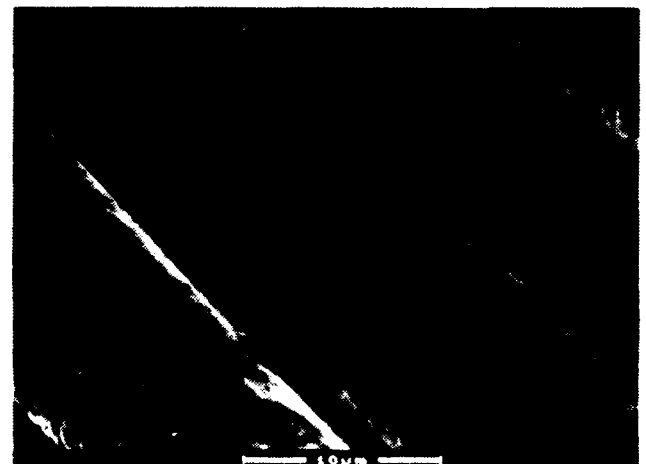


Figure 14. SEM Micrograph of Gr/ZE41A Transverse Fracture Surface.

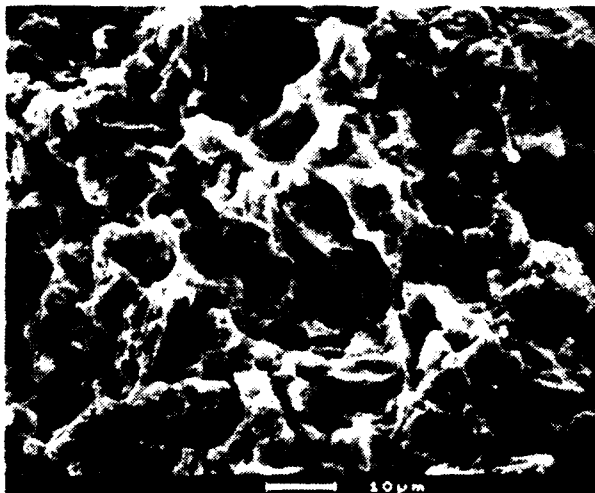


Figure 15. SEM Micrograph of  $B_4C/AZ61A$  Fracture Surface.

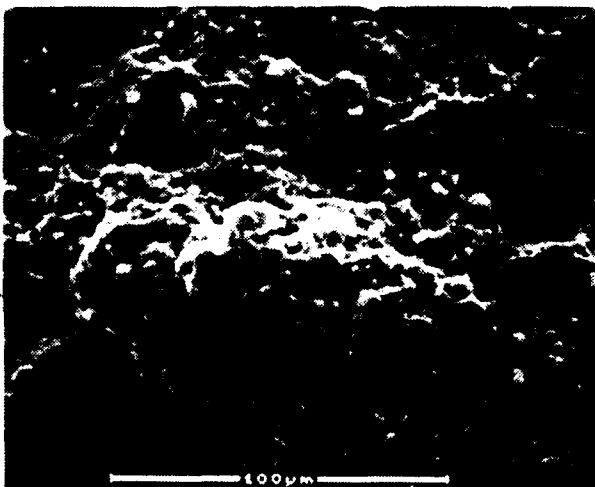


Figure 16. SEM Micrograph of  $SiC_p/ZK60A$  Fracture Surface.

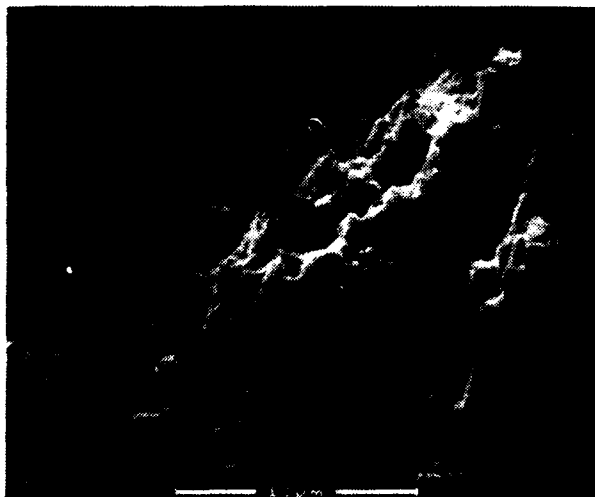


Figure 17. SEM Micrograph of  $SiC_w/ZK60A$  Fracture Surface.

## DISCUSSION

Previous studies on the microstructure and properties of  $\alpha-Al_2O_3/ZE41A$ [9] and  $Gr/AZ91C$ [10] recognized that both materials exhibit near theoretical longitudinal properties. The fracture morphologies of both  $\alpha-Al_2O_3/ZE41A$  and  $Gr/AZ91C$  were consistent with that of a well bonded composite. The key common factor between the two composites appeared to be the interfacial reaction zone. An ultra-fine grain reaction product,  $MgAl_2O_4$  in the case of  $\alpha-Al_2O_3/ZE41A$  and  $MgO$  in the case of  $Gr/AZ91C$ , was present to provide good interfacial shear strength. The spinel phase  $MgAl_2O_4$  was the reaction product from the  $\alpha-Al_2O_3$  and molten magnesium. The  $MgO$  resulted from reaction of the amorphous  $SiO$  coating material and molten magnesium. The mean grain diameter within the reaction zone was less than 500Å. Similarly, in  $B_4C/AZ61A$  where a thin film of fine grain reaction product was present, fracture occurred through particle cleavage rather than interfacial debonding. This may have accounted for the best toughness result of all the composites tested in this study.

Transverse strength and toughness in  $Gr/AZ91C$  were extremely low compared to the  $\alpha-Al_2O_3/ZE41A$  and the theoretical ROM prediction. The weakest link in the  $Gr/AZ91C$  was the unreacted amorphous silicon oxide coating which failed under tension in transverse loading. In  $Gr/ZE41A$ , the large grain rare earth oxide interface ( $>0.1\mu m$ ) formed a thick brittle reaction layer which accounted for the poor longitudinal tensile and toughness properties. The oxide was a reaction product from the amorphous  $SiO$  coating and the rare earths (La and Ce) in molten  $ZE41A$  Mg. A large brittle interface is known to have detrimental effects on fiber/matrix load transfer in metal matrix composites[11]. However, in the transverse direction under constant stress, the  $Gr/ZE41A$  tensile strength and modulus were comparable to  $Gr/AZ91C$ .

The  $SiC_p/ZK60A$  with all its whiskers aligned in the extrusion and loading direction allowed load transfer to the reinforcement as in continuously fiber reinforced composite. Better strength and toughness were achieved with  $SiC_p/ZK60A$  than  $SiC_w/ZK60A$  in the longitudinal direction and the reversed were true in the transverse direction. TEM showed no interfacial reaction in both  $SiC_p/ZK60A$  nor  $SiC_w/ZK60A$ . A clean and coherent interface provide the interfacial bonding. Fracture paths in these composites did follow the interface. Whisker pullouts were evident. Enhancement of the interfacial bond in  $SiC_p/ZK60A$  and  $SiC_w/ZK60A$  could further improve upon its current properties.

In  $B_4C/AZ61A$ , fracture always initiated from surface flaws or oxide inclusions. Fracture origin was not obvious in  $SiC_p/ZK60A$  or  $SiC_w/ZK60A$ . Either the critical flaw size that initiates fracture increases with the reinforcement size or the average defect diameter in the  $SiC/Mg$  was smaller than that of  $B_4C/AZ61A$ . It is conceivable that subject of a more severe extrusion reduction can decrease average defect size as would be in the case of the two  $SiC/Mg$ .

All the  $Gr/AZ91C$  and  $Gr/ZE41A$  toughness data was obtained through Charpy bars sectioned from the 1/2" plates. Sample tensile specimens taken from 1/2" plates had significantly lower properties than specimens from the 1/4" plates. The fracture

morphologies showed variation of structures within each specimen with noticeable amounts of delamination. Thus the  $K_{I0}$  data presented here does not represent that of a sound casting as did with 1/4" plate material. Better toughness can be anticipated with improvements in casting thicker plates.

Finally, what is presented here is a limited selection of all the magnesium composites being pursued today. The cast continuously fiber reinforced composites had demonstrated superior modulus over all the dispersion strengthened composites and would be ideal for stiffness critical applications. A more severe interfacial reaction than the extruded dispersion strengthened materials was noted for the cast composites. Further studies are needed to tailor the interfacial reaction. Better and more consistent strength and toughness were achieved with the particulate and whisker reinforced composites than with the continuous fiber reinforced composites. The aforementioned trend relating properties and microstructure noted in this study certainly is consistent with that reported from continuous and discontinuous aluminum composites. The effects of reinforcement size, geometry, and dispersements on tensile, modulus, and toughness are similar. However, the effects of heat treatment response to mechanical work on magnesium composites should also be investigated.

#### CONCLUSIONS

1. Better strength and toughness were achieved with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A than Gr/AZ91C Mg and Gr/ZE41A.
2. The  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A demonstrated ROM properties with 1/2" plates whereas the properties in Gr/AZ91C decreased significantly from the 1/4" plate to the 1/2" plate.
3. Presence of a thin and fine grain interfacial reaction layer was characteristic of a well bonded composite as was demonstrated in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/ZE41A, Gr/AZ91C, and B<sub>4</sub>C/AZ61A.
4. A clean, coherent interface in SiC<sub>w</sub>/ZK60A and SiC<sub>p</sub>/ZK60A provided sufficient bonding to exhibit good mechanical properties.
5. B<sub>4</sub>C/AZ61A with the larger particle size reinforcement demonstrated lower strength and modulus but better toughness than SiC<sub>w</sub>/ZK60A and SiC<sub>p</sub>/ZK60A.
6. SiC<sub>w</sub>/ZK60A had better strength and toughness than SiC<sub>p</sub>/ZK60A in the longitudinal direction.
7. Better transverse strength and modulus were achieved with SiC<sub>p</sub>/ZK60A than SiC<sub>w</sub>/ZK60A.

#### ACKNOWLEDGMENT

This author wishes to give special thanks to Mr. J. Nunes, Dr. N. Tsangarakis, Mr. R. Pasternak, Mr. P. Smoot, Mr. C. Lane and Mr. A. Zani for their technical advice and assistance in making this paper possible.

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